

## 锌、镉配位聚合物的合成、晶体结构及荧光性质

王庆伟<sup>\*,1</sup> 王亚男<sup>1</sup> 李秀梅<sup>2</sup> 刘 博<sup>1</sup> 孔志国<sup>1</sup>

(<sup>1</sup> 环境友好材料制备与应用省部共建教育部重点实验室(吉林师范大学),四平 136000)

(<sup>2</sup> 通化师范学院化学学院,通化 134002)

**摘要:** 通过水热法合成了 2 个金属-有机配位聚合物  $[\text{Zn}(\text{boba})(\text{bix})]_n$  (**1**) 和  $[\text{Cd}(\text{L1})(\text{L2})]_n \cdot n\text{H}_2\text{O}$  (**2**) ( $\text{H}_2\text{boba}=4,4'$ -(丁烷-1,2-二氧基)-二苯甲酸,  $\text{bix}=1,4$ -双(咪唑基-1-基)苯,  $\text{H}_2\text{L1}=4$ -(羧基甲氧基)苯甲酸,  $\text{L2}=2$ -(4-羟基)-1*H*-咪唑并[4,5-*f*][1,10]菲咯啉)。并对其进行了元素分析、红外光谱、热重和 X-射线单晶衍射测定。配合物 **1** 为二维网状结构,配合物 **2** 为一维双链结构。此外,还研究了它们的荧光性质。

**关键词:** 水热合成; 晶体结构; 锌配合物; 镉配合物

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## Synthesis, Crystal Structure and Fluorescent Properties of Zinc, Cadmium Coordination Polymers

WANG Qing-Wei<sup>\*,1</sup> WANG Ya-Nan<sup>1</sup> LI Xiu-Mei<sup>2</sup> LIU Bo<sup>1</sup> KONG Zhi-Guo<sup>1</sup>

(<sup>1</sup>Key Laboratory of Preparation and Applications of Environmental Friendly Materials  
(Jilin Normal University), Ministry of Education, Siping, Jilin 136000, China)

(<sup>2</sup>Faculty of Chemistry, Tonghua Normal University, Tonghua, Jilin 134002, China)

**Abstract:** Two metal-organic coordination polymers  $[\text{Zn}(\text{boba})(\text{bix})]_n$  (**1**) and  $[\text{Cd}(\text{L1})(\text{L2})]_n \cdot n\text{H}_2\text{O}$  (**2**) ( $\text{H}_2\text{boba}=4,4'$ -(butane-1,4-diyl dioxy)dibenzoic acid,  $\text{bix}=1,4$ -bis(imidazol-1-yl)-benzene,  $\text{H}_2\text{L1}=4$ -(carboxy-methoxy)-benzoic acid,  $\text{L2}=2$ -(4-hydroxyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline) have been hydrothermally synthesized and structurally characterized by elemental analysis, IR spectrum, TG and single-crystal X-ray diffraction. Complex **1** is 2D network structure, complex **2** exhibits 1D double chain structure. Moreover, their luminescent properties have been investigated in the solid state. CCDC: 987090, **1**; 988451, **2**.

**Key words:** hydrothermal synthesis; crystal structure; zinc complex; cadmium complex; fluorescence

## 0 Introduction

The designed synthesis and characterization of metal-organic coordination polymers have achieved considerable progress in supramolecular chemistry and material chemistry<sup>[1-8]</sup>. The increasing interest in this field is justified not only for their particular

beauty and intriguing structural diversities of architecture, but also for their potential applications as catalytic, conductive, luminescent, magnetic, spin-transition, non-linear optical or porous functional materials<sup>[9-15]</sup>. The combination of metal ions with neutral and anionic mixed bridging ligands makes the assembly process more controllable than a single

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\*通讯联系人。E-mail: wqw611223@163.com

ligand. The prospect of introducing the second or more organic ligands into a reaction system provides further impetus for research on metal-organic supramolecular frameworks. The construction of supramolecular architectures through selective and directional non-covalent forces such as hydrogen bonding,  $\pi \cdots \pi$  and  $C-H \cdots \pi$  interactions in metal-organic frameworks arouses considerable contemporary interest due to their potential applications as functional materials.

The hydrothermal technique is well suited to the preparation of crystals of synthetic minerals, new inorganic materials, and organometallic coordination polymers. Of particular interest to us is the construction of transition metal polymers with new structural features by utilizing hydrothermal synthesis. We report here the preparation and crystal structure of the title complexes  $[Zn(boba)(bix)]_n$  (**1**) and  $[Cd(L1)(L2)]_{2n} \cdot nH_2O$  (**2**), which exhibit 2D network structure and 1D double chain structure.

## 1 Experimental

### 1.1 General procedures

All reagents were purchased commercially from Jinan Camolai Trading Company and used without further purification. Elemental analyses (C, H and N) were measured on a Perkin-Elmer 2400 CHN Elemental Analyzer. IR spectrum was recorded in the range of 4 000~400  $cm^{-1}$  on an Alpha Centaur FT/IR Spectrophotometer using a KBr pellet. TG studies were performed on a Perkin-Elmer TGA7 analyzer. The fluorescent studies were carried out on a computer-controlled JY Fluoro-Max-3 spectrometer at room temperature.

### 1.2 Synthesis

$[Zn(boba)(bix)]_n$  (**1**). The title compound was prepared from a mixture of  $Zn(OAc)_2 \cdot 2H_2O$  (0.044 g, 0.2 mmol),  $H_2boba$  (0.131 g, 0.4 mmol),  $bix$  (0.048 g, 0.2 mmol) and  $H_2O$  (10 mL) in a 30 mL Teflon-lined stainless vessel under autogenous pressure at 170  $^{\circ}C$  for 5 d. After cooling to room temperature, yellow block crystals were collected by filtration and washed with distilled water in 43% yield. Anal. Calcd. (%) for

$C_{32}H_{30}N_4O_6Zn$ : C, 60.81; H, 4.78; N, 8.86. Found (%): C, 60.49; H, 4.35; N, 8.54. IR (KBr,  $cm^{-1}$ ): 3 437w, 3 126w, 2 950w, 1605s, 1 553w, 1 507w, 1 423w, 1 384m, 1 304w, 1 250s, 1 168m, 1 109m, 1 047w, 1 014w, 977w, 856m, 784m, 698w, 656m, 509w.

$[Cd(L1)(L2)]_{2n} \cdot nH_2O$  (**2**). The title compound was prepared from a mixture of  $CdCl_2 \cdot 2H_2O$  (0.114 g, 0.5 mmol),  $H_2L1$  (0.098 g, 0.5 mmol),  $L2$  (0.156 g, 0.5 mmol) and  $H_2O$  (10 mL) in a 30 mL Teflon-lined stainless vessel under autogenous pressure at 170  $^{\circ}C$  for 3 d. After cooling to room temperature, pale yellow block crystals were collected by filtration and washed with distilled water in 46% yield. Anal. Calcd.(%) for  $C_{28}H_{20}CdN_4O_7$ : C, 52.80; H, 3.17; N, 8.80. Found (%): C, 52.35; H, 2.95; N, 8.59. IR (KBr,  $cm^{-1}$ ): 3 433m, 3 158w, 1 591s, 1 537w, 1 481w, 1 460w, 1 439w, 1 400m, 1 359w, 1 333w, 1 277w, 1 266m, 1 223s, 1 171m, 1 143w, 1 109w, 1 076w, 1 061w, 977w, 958w, 857w, 837w, 814w, 793w, 785w, 735m, 725w, 642w, 575w, 552w, 470w.

### 1.3 Structure determination

Single crystal diffraction data of **1** and **2** were respectively collected on a Bruker SMART APEX-CCD diffractometer equipped with a graphite-monochromatic  $Mo K\alpha$  ( $\lambda=0.071\ 073$  nm) radiation at room temperature. The structure was solved by direct methods with SHELXS-97 program<sup>[16]</sup> and refined by full-matrix least-squares techniques on  $F^2$  with SHELXL-97<sup>[17]</sup>. All non-hydrogen atoms were refined anisotropically and the hydrogen atoms of organic ligands were generated geometrically. The selected bond parameters are given in Table 1 and 2.

Crystal data for **1**:  $C_{32}H_{30}N_4O_6Zn$ , monoclinic, space group  $P2_1/c$ ,  $M_r=631.97$ ,  $a=1.159\ 22(18)$  nm,  $b=0.777\ 62(11)$  nm,  $c=1.707\ 8(3)$  nm,  $\beta=96.275(3)^{\circ}$ ,  $V=1.530\ 2(4)$  nm<sup>3</sup>,  $Z=2$ ,  $F(000)=656$ ,  $\mu(Mo\ K\alpha)=0.852$  mm<sup>-1</sup>,  $D_c=1.372$  g $\cdot$ cm<sup>-3</sup>, 8 136 reflections measured, 3 008 unique ( $R_{int}=0.052\ 7$ ), 1 960 observed reflections with  $I>2\sigma(I)$ ,  $R=0.049\ 0$ ,  $wR=0.114\ 7$ ,  $S=1.000$ .

Crystal data for **2**:  $C_{28}H_{20}CdN_4O_7$ , triclinic, space group  $P\bar{1}$ ,  $M_r=636.88$ ,  $a=1.004\ 8(5)$  nm,  $b=1.088\ 7(5)$  nm,  $c=1.262\ 9(5)$  nm,  $\alpha=67.681(5)^{\circ}$ ,  $\beta=80.530(5)^{\circ}$ ,  $\gamma=77.325(5)^{\circ}$ ,  $V=1.241\ 8(10)$  nm<sup>3</sup>,  $Z=2$ ,  $F(000)=640$ ,

**Table 1** Selected bond lengths (nm) and bond angles (°) for **1** and **2**

1					
Zn(1)-O(1)	0.193 9(3)	Zn(1)-N(1)	0.201 9(3)	Zn(1)-N(1A)	0.201 9(3)
Zn(1)-O(1A)	0.193 9(3)				
O(1)-Zn(1)-O(1A)	118.17(17)	O(1)-Zn(1)-N(1)	113.60(11)	O(1A)-Zn(1)-N(1)	104.12(11)
O(1)-Zn(1)-N(1A)	104.12(11)	O(1A)-Zn(1)-N(1A)	113.60(11)	N(1)-Zn(1)-N(1A)	102.33(16)
2					
Cd(1)-O(1)	0.247 4(3)	Cd(1)-O(2)	0.232 0(3)	Cd(1)-O(4A)	0.239 7(3)
Cd(1)-O(4B)	0.236 7(3)	Cd(1)-O(5A)	0.267 7(3)	Cd(1)-N(1)	0.232 5(3)
Cd(1)-N(2)	0.233 4(3)				
O(2)-Cd(1)-N(1)	92.06(11)	O(2)-Cd(1)-N(2)	159.15(10)	N(1)-Cd(1)-N(2)	71.72(10)
O(1)-Cd(1)-O(4B)	111.99(10)	N(1)-Cd(1)-O(4B)	151.05(10)	N(2)-Cd(1)-O(4B)	87.54(9)
O(2)-Cd(1)-O(4A)	87.40(10)	N(1)-Cd(1)-O(4A)	129.38(9)	N(2)-Cd(1)-O(4A)	92.88(10)
O(2)-Cd(1)-O(1)	53.89(10)	N(1)-Cd(1)-O(1)	98.62(10)	N(2)-Cd(1)-O(1)	139.63(10)
O(4B)-Cd(1)-O(1)	84.03(9)	O(4A)-Cd(1)-O(1)	120.53(9)		

Symmetry transformations used to generate the equivalent atoms: **1**: A:  $2-x, y, -z+1/2$ ; **2**: A:  $x, y+1, z$ ; B:  $1-x, 1-y, 1-z$ .

**Table 2** Parameters between the planes in **2**

Plane	Distance between ring centroids / nm	Dihedral angle / (°)	Perpendicular distance of plane (I) on ring J / nm	Perpendicular distance of plane (J) on ring I / nm
N3C21N4C14C22(I)→N3C21N4C14C22(J) [A]	0.398 5(3)	0	0.371 39(18)	0.371 39(18)
N3C21N4C14C22(I)→N1C26C25C24C23C27(J) [B]	0.404 0(3)	2.3(2)	0.346 64(17)	0.343 83(16)
N1C26C25C24C23C27(I)→N1C26C25C24C23C27(J) [B]	0.404 4(3)	0	0.341 55(16)	0.341 55(16)
N1C26C25C24C23C27(I)→C13C14C22C23C27C28(J) [B]	0.388 5(3)	0.34(19)	0.342 42(16)	0.342 48(16)
N1C26C25C24C23C27(I)→C15C16C17C18C19C20[J] [A]	0.399 6(3)	8.2(2)	0.340 30(16)	0.344 53(18)
C13C14C22C23C27C28(I)→C15C16C17C18C19C20(J) [A]	0.348 5(3)	8.0(2)	0.346 72(16)	0.343 92(18)

Symmetry transformations used to generate the equivalent atoms: A:  $2-x, 2-y, 2-z$ ; B:  $1-x, 2-y, 2-z$ .

$\mu(\text{Mo } K\alpha)=0.937 \text{ mm}^{-1}$ ,  $D_c=1.703 \text{ g} \cdot \text{cm}^{-3}$ , 6 886 reflections measured, 4 871 unique ( $R_{\text{int}}=0.017$ ), 3 838 observed reflections with  $I>2\sigma(I)$ ,  $R=0.037 6$ ,  $wR=0.076 1$ ,  $S=1.023$ .

CCDC: 987090, **1**; 988451, **2**.

## 2 Results and discussion

### 2.1 IR spectrum

Complex **1**: The  $\text{COO}^-$  is coordinated with its asymmetric and symmetric stretching appearing at  $1\,605$  ( $\nu(\text{OCO})_{\text{asym}}$ ) and  $1\,384 \text{ cm}^{-1}$  ( $\nu(\text{OCO})_{\text{sym}}$ )<sup>[18]</sup>, respectively. The  $\Delta\nu(\nu(\text{OCO})_{\text{asym}}-\nu(\text{OCO})_{\text{sym}})$  is  $266 \text{ cm}^{-1}$  ( $>200 \text{ cm}^{-1}$ ), showing the presence of monodentate linkage of carboxylates in the dianions. Thus the carboxylates coordinate to the metal as monodentate

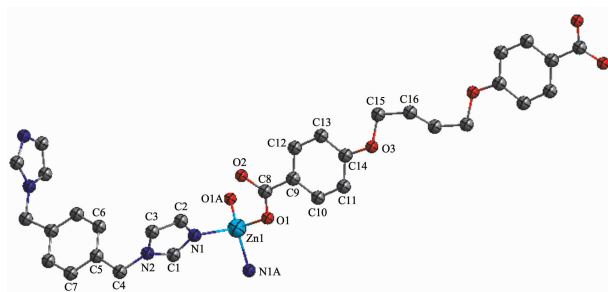
ligands via the carboxylate groups<sup>[19]</sup>.

Complex **2**: Infrared spectroscopy of the title complex shows a strong broad absorption peak at  $3\,433 \text{ cm}^{-1}$ , indicating the existence of water molecules in the coordination polymer. The peak around  $1\,591 \text{ cm}^{-1}$  is assigned to the asymmetric stretching vibration of carboxyl groups, and another strong absorption peak at  $1\,400 \text{ cm}^{-1}$  is due to the symmetric stretching vibration of carboxyl groups. The absence of any strong bands around  $1\,700 \text{ cm}^{-1}$  in the IR spectrum is proven to be deprotonated in the carboxyl group of  $\text{H}_2\text{L1}$ <sup>[20]</sup>.

### 2.2 Description of the structure

Complex **1**: A single crystal X-ray diffraction study reveals that complex **1** exhibits a 2D network

structure. The molecular structure of **1** consists of one  $\text{Zn}^{2+}$  ion, one boba ligand and one bix ligand. As shown in Fig.1, the  $\text{Zn}(\text{II})$  ion is tetra-coordinated by two nitrogen donors (N(1), N(1A, Symmetry code: A:  $2-x, y, -z+1/2$ )) from two flexible bix molecules and two carboxylate oxygen atoms (O(1), O(1A, Symmetry code: A:  $2-x, y, -z+1/2$ )) from two different boba ligands to furnish a distorted tetrahedral coordination geometry. The bond distances of Zn-O in compound **1** are 0.193 9 (3) nm, and Zn-N bond length is 0.201 9(3) nm, which are in the normal range and the coordination angles around Zn atom are in the range  $102.33(16)^\circ \sim 118.17(17)^\circ$ . In the crystal structure of complex **1**, the boba ligands display one kind of coordination mode, namely monodentate bridging mode, and the bix ligand adopts *cis*-conformation bridging mode with a dihedral angle between the two imidazole rings of  $53.52^\circ$ .

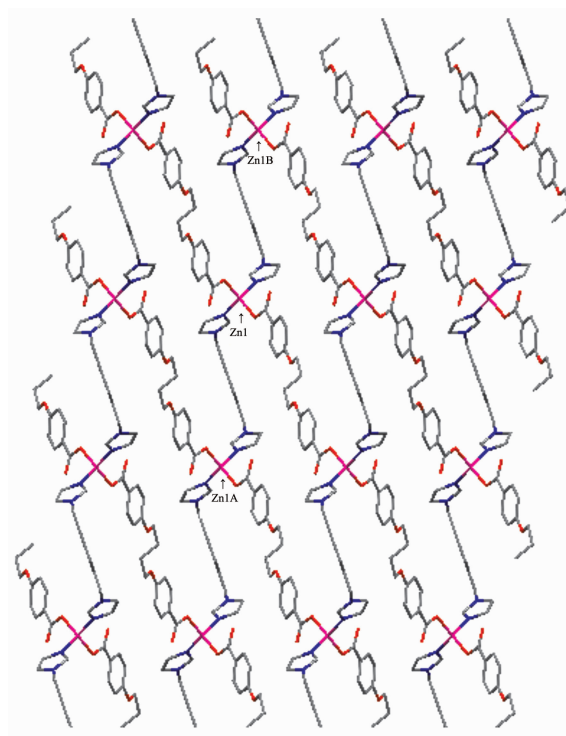


Symmetry code: A:  $2-x, y, -z+1/2$

Fig.1 Coordination environment of  $\text{Zn}(\text{II})$  atoms in **1**

The  $\text{Zn}(\text{II})$  centers forms a type of  $2\text{O}+2\text{N}$  mixed neutrality complex, and are interconnected by the bridging boba ligands to generate an infinite  $\{\text{Zn}_2(\text{boba})_2\}_\infty$  chain along the *b* axis. The  $\text{Zn}\cdots\text{Zn}$  separation through the boba bridges is 1.589 nm, and the neighboring 1D  $\{\text{Zn}_2(\text{boba})_2\}_\infty$  infinite chains are linked via bix ligands to develop into 2D framework. It is noteworthy that there is featuring sixty-six number ring in the two-dimensional network structure along the *b* axis (Fig.2). Furthermore,  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonding interactions between boba molecules and bix ligands ( $\text{C}4\cdots\text{O}2\text{A}$  0.304 85 nm,  $\text{C}16\cdots\text{O}3\text{B}$  0.286 45 nm, symmetric codes: A:  $x, -1+y, z$ ; B:  $1-x, 2-y, 1-z$ ) stabilize the structure of **1**.

Complex **2**: A single-crystal X-ray diffraction

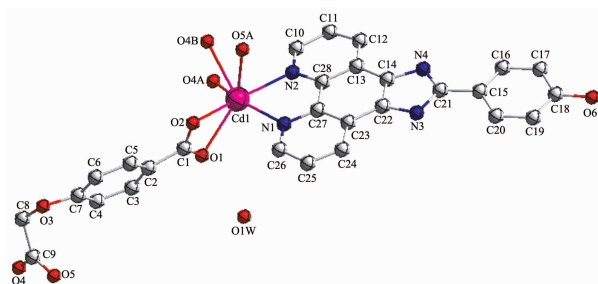


Symmetry codes: A:  $1-x, -y, -z$ ; B:  $2-x, -y, -z$

Fig.2 2D network structure along the *b* axis in **1**

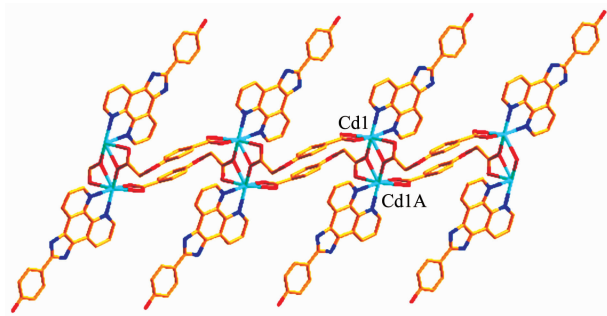
study reveals that the asymmetric unit of complex **2** contains one  $\text{Cd}(\text{II})$  atom, one L1 ligand, one L2 ligand and one crystal water molecule as shown in Fig.3. Cd(1) is seven-coordinated defined by two nitrogen donors (N(1), N(2)) from L2 molecules, five carboxylate oxygen atoms (O(1), O(2), O(4A, Symmetry code: A:  $x, y+1, z$ ), O(4B, Symmetry code: B:  $1-x, 1-y, 1-z$ ), O(5A)) from three different L1 ligands. The bond distances of Cd-O in compound **1** fall in the 0.232 0(3)~0.267 7(3) nm range, and the Cd-N bond lengths are 0.232 5(3)~0.233 4(3) nm, which are very close to those found in another analogous complex  $[\text{Cd}(\text{bpea})(\text{phen})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$  (bpea=biphenylethene-4,4'-dicarboxylate and phen=1,10-phenanthroline)<sup>[20]</sup> and the coordination angles around the Cd atom vary from  $53.89(10)^\circ$  to  $159.15(10)^\circ$ . It is interesting to note that one carboxylate of L1 anion coordinates with one  $\text{Cd}(\text{II})$  atom in a chelating mode, while another carboxylate bridges two  $\text{Cd}(\text{II})$  atoms in a chelating-bridging mode. In these modes, L1 anions link neighboring  $\text{Cd}(\text{II})$  atoms to form a 1D double chain structure (Fig.4). Notably, the L2 ligands are alternately located on both sides of the double chains. More interestingly, the

lateral L2 ligands from adjacent double chains are paired to furnish  $\pi$ - $\pi$  interactions (Table 2), which is similar to the reported compound  $[\text{Mn}(\text{L})(1,4\text{-bdc})]$  ( $\text{L} = 11\text{-fluoro-dipyrido}[3,2\text{-}a':2',3'\text{-}c]\text{phenazine}$  and  $1,4\text{-bdc} = \text{benzene-1,4-dicarboxylate}$ )<sup>[21]</sup>. When these interactions are taken into account, the 1D double chains become a 2D supramolecular layer (Fig.5). Furthermore,  $\text{N-H}\cdots\text{O}$  ( $\text{N3}\cdots\text{O5A}$  0.282 1(4) nm),  $\text{O-H}\cdots\text{N}$  ( $\text{O1W}\cdots\text{N4B}$  0.290 6(5) nm) and  $\text{O-H}\cdots\text{O}$  ( $\text{O1W}\cdots\text{O1}^c$  0.274 8(5) nm,  $\text{O6}\cdots\text{O1WC}$  0.268 1(5) nm,  $\text{O6}\cdots\text{O1}^c$  0.319 7 (4) nm) (symmetric codes: A:  $1-x, 1-y, 2-z$ ; B:  $x, y-1, z$ ; C:  $2-x, 2-y, 2-z$ ) hydrogen bonds further stabilize the structure of **2**.



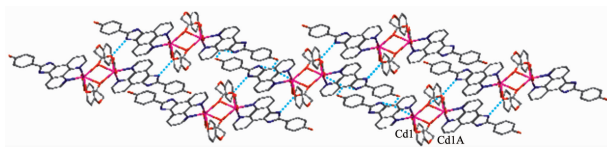
Symmetric codes: A:  $x, y+1, z$ ; B:  $1-x, 1-y, 1-z$

Fig.3 Coordination environment of Cd(II) atom in **2**



Symmetric codes: A:  $1-x, 2-y, 1-z$

Fig.4 View of the 1D double chain structure of complex **2**



Symmetric codes: A:  $1-x, 2-y, 1-z$

Fig.5 2D supramolecular layer structure in **2**

### 2.3 Thermal analysis

TG curve of **1** (Fig.6) show that the first weight loss of 50.98% from 22 to 530  $^{\circ}\text{C}$  corresponds to the removal of boba molecule (Calcd. 51.95%). Upon

further heating, an obvious weight loss (36.23%) occurs in the temperature range of 530 ~770  $^{\circ}\text{C}$ , corresponding to the release of bix ligands (Calcd. 37.71%). After 770  $^{\circ}\text{C}$  no weight loss is observed, which means the complete decomposition of **1**. The residual weight should be ZnO.

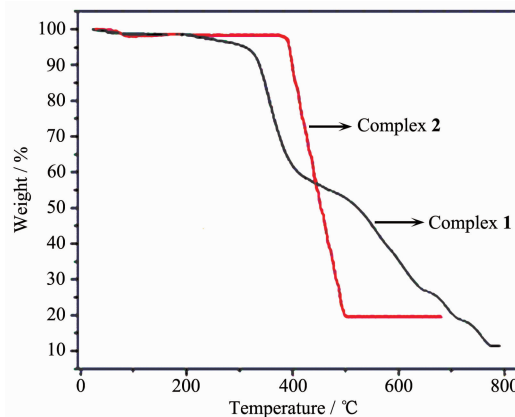


Fig.6 TG curves of complex **1** and **2**

TG curve of **2** (Fig.6) show that the first weight loss of 3.24% from 113 to 132  $^{\circ}\text{C}$  corresponds to the removal of crystal water molecule (Calcd. 2.83%). Upon further heating, an obvious weight loss (78.63%) occurs in the temperature range of 255 ~458  $^{\circ}\text{C}$ , corresponding to the release of L1 and L2 ligands (Calcd. 79.52%). After 458  $^{\circ}\text{C}$  no weight loss is observed, which means the complete decomposition of **2**. The residual weight should be CdO.

### 2.4 Photoluminescent properties

The emission spectrum of complex **1** in the solid state at room temperature is shown in Fig.7. It can be observed that complex **1** exhibits blue photoluminescence with an emission maximum at *ca.* 453 nm upon excitation at 387 nm. In order to understand the nature of these emission bands, we first analyzed the photoluminescence properties of free 4,4'-(butane-1,4-diylidioxy)dibenzoic acid, and confirmed that it does not emit any luminescence in the range of 400~800 nm. And then we investigated the emission spectrum of bix itself and the result indicated that it does not emit any luminescence in the range 400~800 nm, which has also been confirmed previously. Thus, according to the previous literature<sup>[23]</sup>, the emission band could be assigned to the emission of ligand-to-



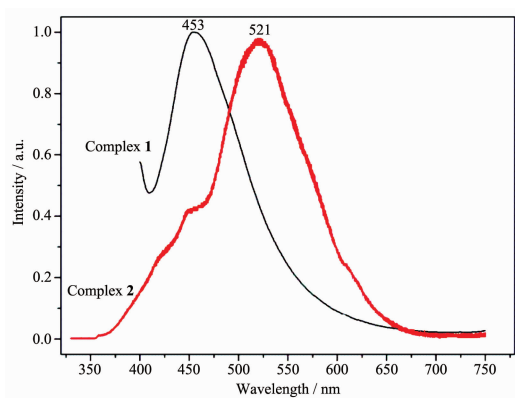


Fig.7 Solid-state emission spectrum of **1** and **2** at room temperature

metal charge transfer (LMCT). For possesses strong fluorescent intensity, it appears to be good candidates for novel hybrid inorganic-organic photoactive materials.

The solid-state photoluminescent properties of H<sub>2</sub>L1, L2 and compound **2** have been investigated in solid state at room temperature. The emission spectra of H<sub>2</sub>L1 and L2 ligands show emissions at 510 nm ( $\lambda_{\text{ex}}=325$  nm) and 551 nm ( $\lambda_{\text{ex}}=325$  nm), respectively, which are probably attributable to the  $\pi^* \rightarrow n$  or  $\pi^* \rightarrow \pi$  transition<sup>[24]</sup>. The emission spectrum for compound **2** (Fig.7) shows a main peak at 521 nm ( $\lambda_{\text{ex}}=325$  nm) that red-shifted for 11 nm with respect to the band shown by H<sub>2</sub>L1 anion. On complexation of L1 anion and L2 ligand with the Cd(II) atom, the emission arising from free L2 was not observed. Therefore, the emission of compound **2** can be assigned to the emission of H<sub>2</sub>L1 ligand.

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